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TA SPUTTERING TARGET AND ITS MANUFACTURING METHOD

Inventors:	Fumiyuki Shimizu Nikko Kinzoku K.K. Hitachi Refinery 3453 Miyata-cho, Hitachi-shi, Ibaraki-ken
	Toshihiro Yano Nikko Kinzoku K.K. Hitachi Refinery 3453 Miyata-cho, Hitachi-shi, Ibaraki-ken
	Yasushi Umemoto Nikko Kinzoku K.K. Hitachi Refinery 3453 Miyata-cho, Hitachi-shi, Ibaraki-ken

Applicant:

592258063
Nikko Kinzoku K.K.
2-10-1 Toranomom, Minator-ku,
Tokyo

Agent:

Takeshi Imai,
patent attorney

Abstract

Objective

To establish a means to obtain a uniform, high-performance thin film in a stable manner from sputtering using a Ta sputtering target.

Constitution

A Ta sputtering target is constituted by a plastic processing material of Ta prepared by melting with a total gas component content of less than 100 ppm and an average crystal particle diameter of less than 1 mm. Furthermore, for the manufacture of this Ta sputtering target, after cold forging of a Ta ingot with a total gas component content of less than 100 ppm at a processing ratio of more than 90%, a process is adopted in which a heat treatment is carried out in a vacuum less than 0.1 mmbar at a heating temperature of 900-1300°C to cause recrystallization.

Claims

1. A Ta sputtering target characterized by the fact that it consists of a plastic processing material of Ta prepared by melting with a total gas component content of less than 100 ppm and an average crystal particle diameter of less than 1 mm.
2. A method for the manufacture of the Ta sputtering target described in Claim 1, characterized by the fact that it includes a process in which a heat treatment is carried out in a vacuum less than 0.1 mmbar at a heating temperature of 900-1300°C to cause recrystallization, after cold forging of a Ta ingot with a total gas component content of less than 100 ppm at a processing ratio of more than 90%.

Detailed explanation of the invention

[0001]

Industrial application field

The present invention relates to a Ta (tantalum) sputtering target appropriate for the manufacture of semiconductor devices, the coating of thermal ray absorption glass for automobiles and the like, as well as its manufacturing method.

[0002]

Conventional technology and its problems

Conventionally, a SiO_2 film has been used as an insulating film between electrode wiring layers in a semiconductor device. However, partly due to dissatisfaction with the SiO_2 film owing to the high integration of LSI in recent years, attempts to use a Ta_2O_5 thin film instead of the SiO_2 film have proceeded.

[0003]

In the formation of this Ta_2O_5 thin film, a CVD method using an organic reaction gas and a sputtering method for the sputtering of a Ta sputtering target (to be simply called "Ta target" hereafter) in an "argon-oxygen mixed gas" have been used. From the overall viewpoint, the sputtering method is more advantageous.

[0004]

As electrodes of VLSI, Mo, W, and other high melting point metal silicide thin films have been used to date. However, in recent years, the Ta silicide film has drawn attention. It has been considered as a promising electrode material for the future. In the formation of a Ta silicide film, several methods can also be considered. As one of them, a Ta film is adhered to polycrystal silicon by sputtering using a Ta target and then silicon and Ta are reacted to form a Ta silicide film.

[0005]

Furthermore, recently, attempts have been made to use thin films of Ta as thermal ray absorption films by coating them on the surface of automotive glass, etc. Even from this respect, an increase in demand for Ta targets is expected.

[0006]

However, at present the previously mentioned Ta target used for the formation of a thin film of "Ta," "Ta silicide" or the like has been manufactured by a method in which metal Ta

obtained industrially by an electrolysis process or the like is melted to form a Ta ingot and this is processed to the desired shape and dimensions.

[0007]

As described previously, the application field has grown remarkably for thin films of "Ta" and "Ta alloys". However, according to detailed investigations by the present inventors, in the case of sputtering, the "Ta" or "Ta alloy" thin films formed lack uniformity. There has been a concern about this causing considerable adverse effects on the performance of semiconductor devices, thermal ray absorption glass, and so on.

[0008]

As a result, the objective of the present invention is to establish a means to eliminate the previously mentioned problems of sputtering using a Ta target and to obtain a uniform, high-performance thin film in a stable manner.

[0009]

As a result of zealous investigation to achieve the objective mentioned previously, the present inventors have achieved the following discoveries.

A) The crystal particle size of the Ta target used has a major effect on the uniformity of the thin film formed by sputtering using a Ta target. There is a tendency for a small crystal particle size to be preferred. In particular, if the average crystal particle diameter is adjusted to less than 1 mm, the uniformity improvement effect of the thin film formed is remarkable.

[0010]

B) During the process of detailed investigations, it has been found that the "crystal particle microfining of the Ta target" is surely effective in achieving uniformity of the thin film formed. However, by the microfining of the crystal particles alone, there is a limit in uniformity improvement of the thin film mentioned previously. Even if the average crystal particle diameter of the Ta target is less than 1 mm, there have been cases during film formation on an industrial scale in which it was difficult to form steadily and stably thin films that are sufficiently uniform for use in a semiconductor device, thermal ray absorption glass or the like.

[0011]

C) However, if the total content of O, N, C, S, H, and other gas components in the Ta target is decreased to less than 100 ppm which is unparalleled in industrial products and the average crystal particle diameter is adjusted to less than 1 mm, the uniformity of the thin film

formed will be improved to a large extent. Even in film formation operation on an industrial scale, adverse effects on the performance of a semiconductor device, thermal ray absorption glass and other products caused by the "nonuniformity of the thin film obtained" can be inhibited to a practically negligible level.

[0012]

D) The previously mentioned Ta target with the total content of gas components less than 100 ppm can be manufactured by using, as a starting material, a "Ta cast ingot" obtained by the "electron beam cold hearth remelt method" in which, for example, a feedstock melt obtained by electron beam melting is maintained inside a water-cooled cold hearth to cause impurities to escape in a vacuum environment, the melt overflows into a mold, and it is withdrawn from underneath while it continuously solidifies.

E) Furthermore, the Ta target with a microfine structure having an average particle diameter of less than 1 mm can be realized by the implementation of forced cold processing of the "Ta melt material" with a low gas component content mentioned previously, and the implementation of a recrystallization heat treatment of this under specific conditions.

[0013]

The present invention has been accomplished on the basis of the discovered items mentioned previously. It has a major characteristic in the "aspect that the Ta target is constituted by a plastic processing material of Ta prepared by melting with a total gas component content of less than 100 ppm and an average crystal particle diameter of less than 1 mm." Furthermore, it is also characterized by the fact that "after cold forging of a Ta ingot with a total gas component content of less than 100 ppm under the conditions of a processing ratio of more than 90%, by the adoption of a heat treatment in a vacuum at less than 0.1 mmbar and a heating temperature of 900-1300°C to cause recrystallization, a Ta target with a total gas component content of less than 100 ppm and an average crystal particle diameter of less than 1 mm described previously can be manufactured in a stable manner."

[0014]

Here, the gas component content in the Ta target is restricted to less than 100 ppm total because, if the total gas component content is less than 100 ppm in particular, the amount of the gas released during usage in a high vacuum will decrease drastically and particle generation will practically disappear. If the total gas component content is less than 100 ppm for the Ta target with an average crystal particle diameter of less than 1 mm, the uniformity (film thickness and film characteristics) of the thin films formed by sputtering will improve to a large extent and the

stability of the film formed will also improve remarkably. It has been described previously that it is acceptable to use the "Ta cast ingot" obtained by the "electron beam cold hearth remelt method" as a starting material in order to obtain a Ta target with a total gas component content of less than 100 ppm. However, if possible, it is desirable to decrease the various gas components or other impurity components contained in the target to the following levels:

Gas components

O: less than 50 ppm, N: less than 50 ppm, C: less than 50 ppm, S: less than 10 ppm, and H: less than 10 ppm.

Other impurity components

Nb: less than 0.01 wt%, W: less than 0.05 wt%, Fe: less than 0.01 wt%, Al: less than 0.01 wt%, and Ni: less than 0.01 wt%.

[0015]

Furthermore, the average crystal particle diameter of the Ta target is restricted to less than 1 mm because, as described previously, if the average crystal particle diameter exceeds 1 mm, even if, for example, the gas component content in the Ta target is less than a total of 100 ppm, the uniformity of the thin film formed by sputtering is insufficient, and the adverse effects on the performance of a semiconductor device, thermal ray absorption glass and other products cannot be eliminated. Furthermore, the Ta target is a "melt material" because the microfine crystal particle structure or the gas component content described previously cannot be achieved with powder metallurgy material.

[0016]

During the manufacture of the Ta target, first of all, the Ta ingot is subjected to cold forging at a processing ratio of more than 90% because, if said processing ratio is less than 90%, uniform crystal particles with an average particle diameter of less than 1 mm cannot be obtained even if a recrystallization heat treatment is carried out.

[0017]

Moreover, the recrystallization heat treatment conditions are restricted in the manner described previously because of the following reasons.

a) Heating environment

If the degree of vacuum of the heat treatment is more than 0.1 mmbar, the surface of the material to be treated will be contaminated by oxygen, nitrogen and so on. There will be adverse effects on the yield of the material and the performance of the thin film obtained by sputtering.

b) Heating temperature

If the heating temperature is under 900°C, sufficient recrystallization cannot be achieved and a uniform, microfine crystal particle structure cannot be obtained. On the other hand, if heating is above 1,300°C, coarsening of the crystal particles will occur and a uniform, microfine crystal particle structure cannot be obtained. From the material subjected to the cold forging-recrystallization heat treatment under the conditions mentioned previously, the Ta targets are cut out mechanically.

[0018]

In Ta ingot to be supplied for cold forging, the material prepared by melting so that the total gas component content is less than 100 ppm is used. In order to obtain industrially such a high-purity Ta ingot, the "electron beam cold hearth remelt method" can be used. This "electron beam cold hearth remelt method", as exemplified in Figure 1, is a melting-casting method in which a water cooled cold hearth (3) is installed in front of a water cooled copper mold (2) installed inside the melting chamber (1) of an electron beam melting facility. "The feedstock melt obtained by melting a feedstock electrode (5) supplied from the feedstock horizontal charging apparatus (4) with electron beams from the electron gun (6)" is maintained inside the cold hearth (3) and it overflows. It is cast in the water cooled copper mold (2). While it is being solidified continuously, it is withdrawn from underneath as an ingot (7). In the diagram, the symbol (6') shows a warmth-maintaining electron gun, and the symbol (8) shows a vacuum pump.

[0019]

In this method, the melt obtained by electron beam melting is cast inside the mold while it is allowed to reside in the cold hearth for an appropriate time. Therefore, during residence inside the cold hearth, easily volatilized impurities are sufficiently volatilized into the vacuum environment and removed to obtain a high-purity ingot. Here, the present inventors, who have observed the excellent high-purification effect of the "electron beam cold hearth remelt method," have confirmed that gas components and other impurities can be decreased sufficiently to "an extent such that essentially no gases are generated, even if it is used at high temperature under a high vacuum," even though the Ta melt material is obtained using a one-pass melting operation) after the repetition of manufacturing tests for the melt material by the application of said method

to the melting-casting of Ta. Furthermore, if melting is carried out so that the total gas component content in the Ta melt material is less than 100 ppm by this method, it has been found that other impurities (alkali metals, radioactive elements and so on) to be avoided in the Ta target can also be reduced to a level tolerable for use as a semiconductor device. These discoveries have made a major contribution to the accomplishment of the present invention. As feedstock to be supplied for electron beam cold heart remelting, Ta powder or Ta scrap is acceptable.

[0020]

In continuation, the present invention will be further explained specifically with an application example.

Application example

First of all, a tube (outside diameter 120 mm x length 800 mm x thickness 1 mm) made of commercially pure Ti was filled with Ta scrap, and commercially pure circular disks were TIG welded on both open ends of the tube to prepare a melt electrode material. The compositional analytical values of the Ta scrap used here are shown in Table I.

[0021]

Table I

		① 化 学 成 分 (重量割合)										
		%						ppm				
		Al	Fe	Ti	W	Mo	Nb	O	N	C	S	H
②	Ti ₂ -埋入Ta電極	0.001	0.005	3.7	10	20	2.0	2700	55	140	1	30
③	Ta溶製鑄塊	0.0003	<0.001	<0.001	10	15	2.0	30	12	27	<1	<1

Key: 1 Chemical composition (weight contents)
 2 Ta electrode made of Ti tube
 3 Ta melt ingot

[0022]

Next, the melt electrode material described previously was melted with the electron beam cold hearth remelting facility as shown in Figure 1 under the conditions of

Pressure inside the melting chamber: 10^{-4} mmbar,

Electron beam output: 400 kW,

Melting temperature: 3200°C ,

Surface area of the melt inside the cold hearth: 500 cm^2 ,

Casting rate: 50 kg/hr.

It was then cast in a water cooled copper mold to make a Ta ingot. A compositional analysis was carried out for the Ta ingot obtained in this manner. The results are also shown in Table I. As shown in this Table I, it was found that Ti prone to volatilization loss was removed by evaporation by the electron beam cold hearth remelting, and a high-purity Ta ingot was prepared.

[0023]

Next, the previously mentioned Ta ingot obtained was subjected to cold (room temperature) forging at a pressing ratio of 95% to obtain a plate material. This was then subjected to mechanical processing. After the finishing of the surface had been carried out and the target shape and dimensions had been obtained, a vacuum heat treatment was carried out under a vacuum of 10^{-4} mmbar and at $1,100^{\circ}\text{C}$ for 2 h, to cause recrystallization. The total of the gas component contents is shown in Table I. A Ta target having less than 100 ppm in the same manner as the Ta ingot and a uniform structure with an average crystal particle diameter of $500\text{ }\mu\text{m}$ was manufactured.

[0024]

A sputtering test onto a glass surface was carried out using the Ta target manufactured in this manner. There was no particle generation, and a Ta thin film extremely uniform in both film thickness and film quality formed.

[0025]

Summary of the effect

As explained previously, according to the present invention, Ta sputtering targets capable of forming thin films more uniform than conventional products can be supplied in a stable manner. A major contribution to an improvement in the performance of parts utilizing Ta or Ta alloy thin films can be expected. An extremely useful effect in the industry can be achieved.

Brief description of the figures

Figure 1 is a schematic illustrative diagram of an example of the electron beam cold hearth remelt apparatus that can be used in the method of the present invention.

Figure 2 is a schematic illustrative diagram for a melt electrode material used in an application example.

Explanation of symbols

- 1 Melting chamber
- 2 Water cooled copper mold
- 3 Cold hearth
- 4 Feedstock horizontal charging apparatus
- 5 Feedstock electrode
- 6 Electron gun
- 6' Electron gun
- 7 Ingot
- 8 Vacuum pump

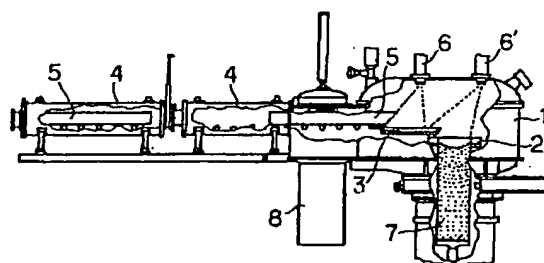


Figure 1

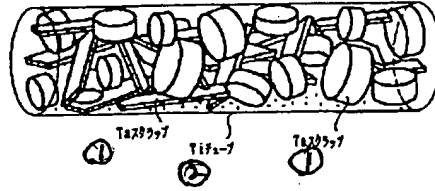


Figure 2

Key: 1 Ta scrap
2 Ti tube